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NATIONAL WATER QUALITY NETWORK OPERATING MANUAL. A GUIDE FOR LABORATORIES PARTICIPATING IN SAMPLING AND ANALYTICAL ACTIVITIES OF THE NATIONAL WATER QUALITY NETWORK

Robert A. Taft Sanitary Engineering Center
Cincinnati, Ohio

October 1960

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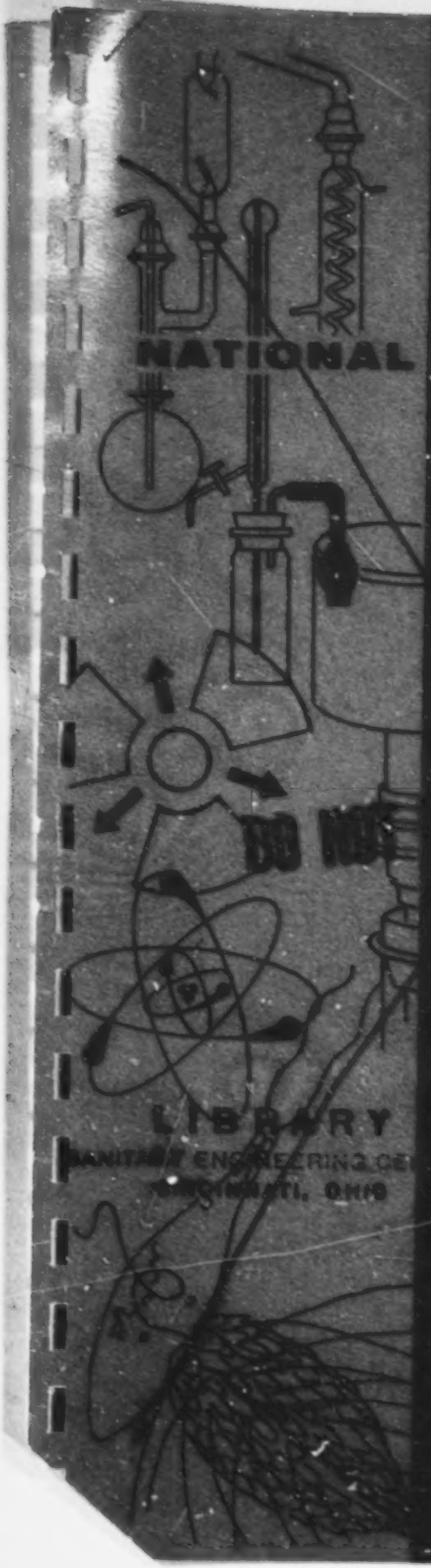

NATIONAL WATER QUALITY NETWORK
operating manual

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Laboratories

U.S. DEPARTMENT OF
HEALTH, EDUCATION, AND WELFARE
Public Health Service

Revised October, 1960

PB 217 663

NATIONAL WATER QUALITY NETWORK

OPERATING MANUAL

A Guide for Laboratories Participating in Sampling
and Analytical Activities of the
National Water Quality Network

Revised October 1960

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U.S DEPARTMENT OF HEALTH, EDUCATION, AND WELFARE
Public Health Service
Bureau of State Services
Division of Water Supply and Pollution Control
Basic Data Branch

Robert A. Taft Sanitary Engineering Center
Cincinnati 26, Ohio

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SECTION I

THE NATIONAL WATER QUALITY NETWORK

THE NATIONAL WATER QUALITY NETWORK was established by the Public Health Service in 1957 in cooperation with State, local and other Federal agencies having related responsibilities for the collection, interpretation and dissemination of basic data on chemical, physical and biological water quality as it relates to water pollution prevention and control.

The overall objectives of the Network are to provide:

a. Information pertaining to the effect of changes in water use and development on water quality at key points in river systems.

b. Continuing information on the nature and extent of pollutants affecting water quality.

c. Data which will be useful in the development of comprehensive water resources programs.

d. Data which will assist State, interstate and other agencies in their water pollution control programs and in the selection of sites for legitimate water uses.

As of October 1960, there were 75 sampling locations in the Network. Each location satisfies one or more of the following criteria:

a. Major waterway used for public water supply, propagation of fish and wildlife, recreational purposes and agricultural, industrial and other legitimate uses.

b. Interstate, coastal and International boundary waters.

c. Waters on which activities of the Federal Government may have an impact.

With its selection as one of the network of key stations, the cooperating laboratory occupies a unique position. Data collected at this and other Network stations will serve as a foundation for the evaluation of water pollution control progress, both on a State and National level. The data will also reflect trends in water quality over the years, thus providing an intelligence service on the quality of the water resources of the Nation. Finally, the cooperating laboratory will be a connecting link or reference point in intrastate or river basin pollution abatement programs.

LABORATORY ANALYSES

The analyses to be carried out have been selected on the basis of emerging and foreseeable problems in water quality management, as described below.

Radioactivity. Interest is rapidly growing in routine monitoring of water for radioactivity. With the development of nuclear power reactors and other peacetime atomic energy uses this interest will become even more intense. State and interstate agencies, and municipalities are becoming increasingly concerned about establishing such programs. Experience with this Network will, it is hoped, encourage and stimulate more effective State and local programs in radiation monitoring.

Organic Chemicals. Studies conducted during the past decade have shown that products of our chemical technology, particularly organic contaminants, are being recovered from waterways in more and more locations as chemical plants increase in size and number, and as their products are increasingly used in the home, in industry, and in agriculture. Synthetic detergents, pesticides of all kinds, and other petro-chemicals are examples of these contaminants. An increasing number of reports of water damages from such pollutants is occurring. Damages which can occur from trace amounts of contaminants include taste and odor, off-tastes in fish, fish kills, foaming, water treatment difficulties, and interference with recreational uses of water. It is important to assess the kinds and quantities of such wastes in our surface waters for the protection of all legitimate water uses.

The measurement of the trace concentrations of organic contaminants requires the use of special techniques since our common procedures lack the needed sensitivity and specificity. Concentration of the contaminants by means of a carbon filter followed by solvent elution and organic analyses presently offers our best means of measuring the segment of organics of most interest. The chloroform and alcohol extractable materials along with separated groups provide a tangible material that can be analyzed and stored for future reference.

Biological Populations. Plankton frequently cause taste, odor, clogging of filters and other difficulties in treatment of water. Plankton are the source of food for higher forms in the aquatic environment. Finally, plankton productivity is directly related, through oxygen production as well as numbers, to the degree of organic enrichment of the streams, as contributed by human wastes and industrial wastes of sewage-like character. Accordingly, a continuous record of plankton populations provides information of importance to water treatment plant operators and to conservation and water pollution control agencies.

Coliforms. The presence of these organisms in water is direct evidence of the presence of sewage. The degree of contamination of the water by these organisms will be of interest to the municipality using the stream for its source of water. The delayed membrane filter technique will be used to carry out this test, as it is the most reliable method available whereby samples can be shipped long distances without deterioration.

Chemical and Physical Parameters. There are many standard chemical and physical measurements which can be made on water. Those of significance to the establishment of water quality vary with the purposes for which the water is to be used. The determinations selected below are of basic value to most water uses; therefore they have been included in the Network program. In certain locations other determinations, of local significance, may be added as the program develops. The determinations to be made at each Network station are:

Alkalinity (or acidity)
Ammonia Nitrogen
Biochemical Oxygen Demand
Chemical Oxygen Demand
Chlorides
Chlorine Demand
Color
Dissolved Oxygen
Hardness
Hydrogen Ion Concentration (pH)
Phosphate (ortho plus poly)
Sulfate
Temperature
Total Dissolved Solids
Turbidity

Many of the cooperating laboratories are performing most of these tests routinely, but a few of them may need to expand their routine to include some of these analyses. The Public Health Service will assist individual laboratories in every way possible, in order that these determinations will be available from each network station. All data collected at each sampling point will be freely exchanged between the Public Health Service, cooperating laboratory and the State agency responsible for water pollution control.

Reference Samples. In a National program where analytical results originate from a multitude of laboratories under different jurisdictions, it is essential that laboratory data be comparable as to precision and accuracy. The Reference Sample activity is a mechanism by which this objective can be achieved. Reference samples will be submitted to each of the collaborating laboratories periodically to assure continued precision and accuracy of all data submitted. Past experience with several Federal and State laboratories has shown that this mechanism leads to increased proficiency of all participants. In addition, it has served to point up routine errors within a laboratory which would otherwise have gone unnoticed.

SAMPLING

All samples are to be of raw waters. All samples are to be collected from the flowing stream or from the raw water intake prior to any treatment, including sedimentation, prechlorination, etc. Samples for determination of radioactivity, organic chemicals, plankton populations, and coliforms are to be shipped to the Public Health Service at Cincinnati for analysis. The chemical analyses will be performed by the cooperating laboratories on samples collected at the same time. Procedures for collection, shipment, and analyses of samples are given in subsequent sections of this manual.

All sampling containers, shipping cases, and other materials will be provided and all shipping charges will be paid by the Public Health Service. Carbon filter units, packed with carbon, will be furnished in individual shipping containers. Containers and shipping cases will be provided for the remaining samples.

Cooperating laboratories are encouraged to develop laboratory competence in the analyses to be made at Cincinnati. The Public Health Service will furnish all possible assistance through training, methods, consultation, reference samples, loan of equipment, and other resources which may be available.

SECTION II

GENERAL INSTRUCTIONS

Routine collection of samples for shipment to the Sanitary Engineering Center will be carried out according to detailed instructions for collection and shipment presented in subsequent sections of this manual. The program includes the following:

a. Carbon Filter - monthly: Start filtration on the day that a sample is collected for the chemical and physical analyses.

b. Plankton Populations - semi-monthly: One $5\frac{1}{2}$ pint sample. Collect on the day that a sample is collected for the chemical and physical analyses.

c. Chemical Oxygen Demand - weekly and monthly: Although this determination is to be performed weekly by the cooperating laboratory as a part of the conventional chemical and physical analyses, a duplicate sample may be furnished to the SEC once per month. This sample should be measured out from the sample of water collected for the B.O.D. analysis. Space has been provided in the carbon filter container for shipment of this sample. Details will be found in Section VII.

d. Coliforms - weekly: This sample should be collected on that day in which a sample is collected for the chemical analysis. Membrane filtration and mailing of this sample to SEC should be done as quickly as possible after collection.

e. Radioactivity - weekly: A one-liter raw water sample should be taken weekly, on the same day a sample is collected for the chemical analysis.

SAMPLING SCHEDULES

Weekly and monthly sampling schedules should be set by each participating laboratory to best fit in with normal routine operations. Once the schedule is established, however, it should be followed as closely as plant operations permit. Any day of the week, except Thursday, is satisfactory. Thursday samples arrive too late for coliform determination. A suggested sampling schedule is as follows:

First sample day of each month:

1. Collect chemical analysis sample. Analyze and mail report forms.

2. Collect, filter and mail the membrane filter coliform sample.

3. Collect raw water radioactivity sample. Mail to SEC.
4. Start the carbon filter run.
5. Collect and ship the plankton sample.

Second sample day of each month:

1. Same as first sample day.
2. Same as first sample day.
3. Same as first sample day.
4. Collect C.O.D. sample and place in the carbon filter shipping case.

Third sample day:

1. Same as first sample day.
2. Same as first sample day.
3. Same as first sample day.
4. Remove carbon filter and ship to SEC. (This may be done any day during the second week, when the carbon filter run may be completed.)
5. Collect and ship plankton sample.

Fourth sample day:

1. Same as first sample day.
2. Same as first sample day.
3. Same as first sample day.

Fifth sample day:

If the month has five sample days, repeat the fourth week's schedule. If a holiday should fall on a sample day, begin the sampling on the first workday following the holiday.

SHIPPING INSTRUCTIONS

All samples, except the carbon filter samples, are to be shipped by mail or parcel post to:

Director
Robert A. Taft Sanitary Engineering Center
U. S. Public Health Service
4676 Columbia Parkway
Cincinnati 26, Ohio

Attention: Water Quality Section

The carbon filter samples are to be shipped by Railway Express, Charges Collect, to the above address. Addressed franked mailing labels and mailing tubes will be provided for each sample shipment.

ASSISTANCE WITH SAMPLING, LABORATORY, AND OTHER OPERATIONAL PROBLEMS

If questions or difficulties arise in connection with the directions and procedures outlined in this manual, the participating agency should contact the Sanitary Engineering Center in Cincinnati, Ohio, by telephone (collect) or by other means of communication, as dictated by the urgency involved. Requests for assistance are to be directed to:

Stephen Megregian, In Charge
Laboratory Activities
Water Quality Section

TRinity 1-1820

SECTION III

CARBON FILTER SAMPLING

The carbon filter sampling operation consists of the passage of up to 5000 gallons of raw water at a rate of 1/4 to 1/2 gallon per minute through the filter. Following the sample run, the filter is shipped to the SEC for analysis.

INSTALLATION OF SAMPLING EQUIPMENT

The carbon filter consists of a piece of Pyrex glass pipe 3 inches in diameter and 18 inches in length. The ends are fitted with brass plates and 3/4-inch galvanized nipples. A stainless steel screen is fixed in a neoprene gasket at both ends. The filter unit will arrive packed with activated carbon ready for use. The special shipping container provided should be used when returning the filter. The unit, with the shipping container is illustrated below.

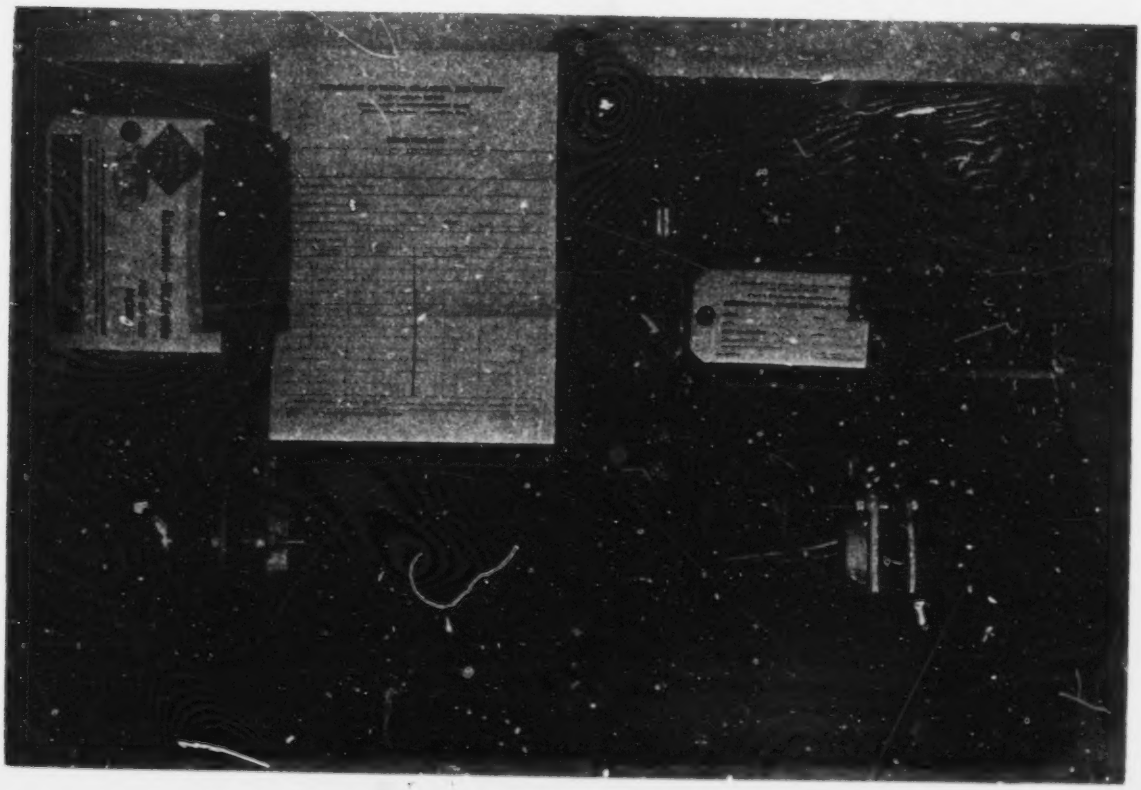

Figure 1 - Carbon Filter Unit and Shipping Container. Note COD Sample Bottle.

PRESETTLING, PREFILTER, AND BACKWASH

River waters will frequently clog the carbon filter before the desired volume of water has been sampled. To prevent this, it is necessary to remove sufficient turbidity to permit the required amount of water to pass through the unit. This may require a presettling tank and a prefilter containing sand and gravel. For waters averaging less than 100 ppm. of turbidity a presettling tank is generally not required.

Presettling Tank. A standard hot water tank connected with the inlet at the bottom and outlet at the top, with a clean-out tap at the bottom, can serve as a presettling tank. The outlet connects to the prefilter containing sand and gravel. The hot water tank should be flushed at frequent intervals to prevent accumulation of solids. Open settling tanks can be used, if flow-thru time at 1/2 gpm is less than 4 hours. With open tanks a pump will be required to move the water through the filter.

Sand Prefilter. The sand prefilter which is provided by the Public Health Service consists of a 3 feet long steel pipe 3 inches in diameter, threaded at both ends, and equipped with 3 inch by 1 inch reducer couplings. Two discs of stainless steel screen are fitted to the inside diameter of the pipe. The space between the screens is packed with 6 inches of 1/8 inch gravel, 24 inches of 0.6 to 0.8 mm sand, and another 6 inches of 1/8 inch gravel. No free space is left in the pipe. The gravel is packed by jarring the pipe while filling. This arrangement provides a strainer rather than a filter with a movable bed. By such an arrangement backflushing can be done without disturbing the filter. The construction details are shown in Figure 2 on Page 10.

FILTER ARRANGEMENT

The presettling tank, the sand prefilter and the carbon filter should be installed at the most convenient source of raw water. If less than 15 psi pressure is available, it may be necessary to pump the water through the system. A drawing of a workable system is shown on Page 11. Exact lengths of pipes, etc. are not given, since these will vary with the local situation. Both the sand and carbon filters are connected with unions at both ends for easy removal. A typical installation is shown in the photograph on page 12.

The raw water flows upward through the sand prefilter and the carbon filter. When the rate of flow through the system falls below 1/4 gpm, backwashing of the sand filter becomes necessary, using a high pressure source of water. A clean water hose is connected to the top of the sand filter, the valve to the carbon filter is closed, the drain valve on the sand filter is opened. The sand is backflushed until the water coming out is clear. The length of time between backwashings will vary. On the Missouri River, for example, it has been found that once every 8 hours is usually sufficient. After backwashing connect the system as before and continue the sampling. To prevent a cross-connection, be sure to disconnect hose at top of sand filter after backwashing. A pressure gauge is inserted in the system to indicate when clogging is taking place in the carbon filter. Total pressure in the filter should not exceed 50 psi.

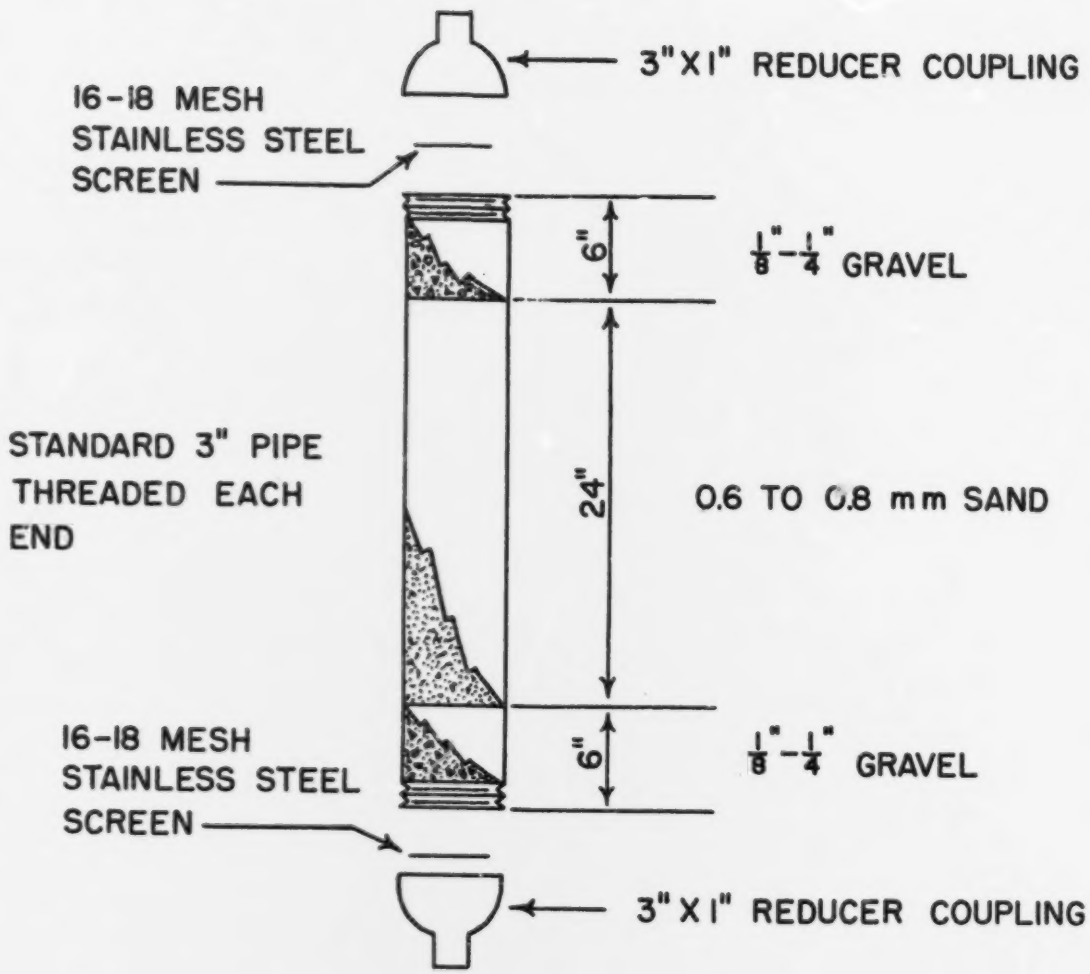

Figure 2 - Details of Sand Prefilter

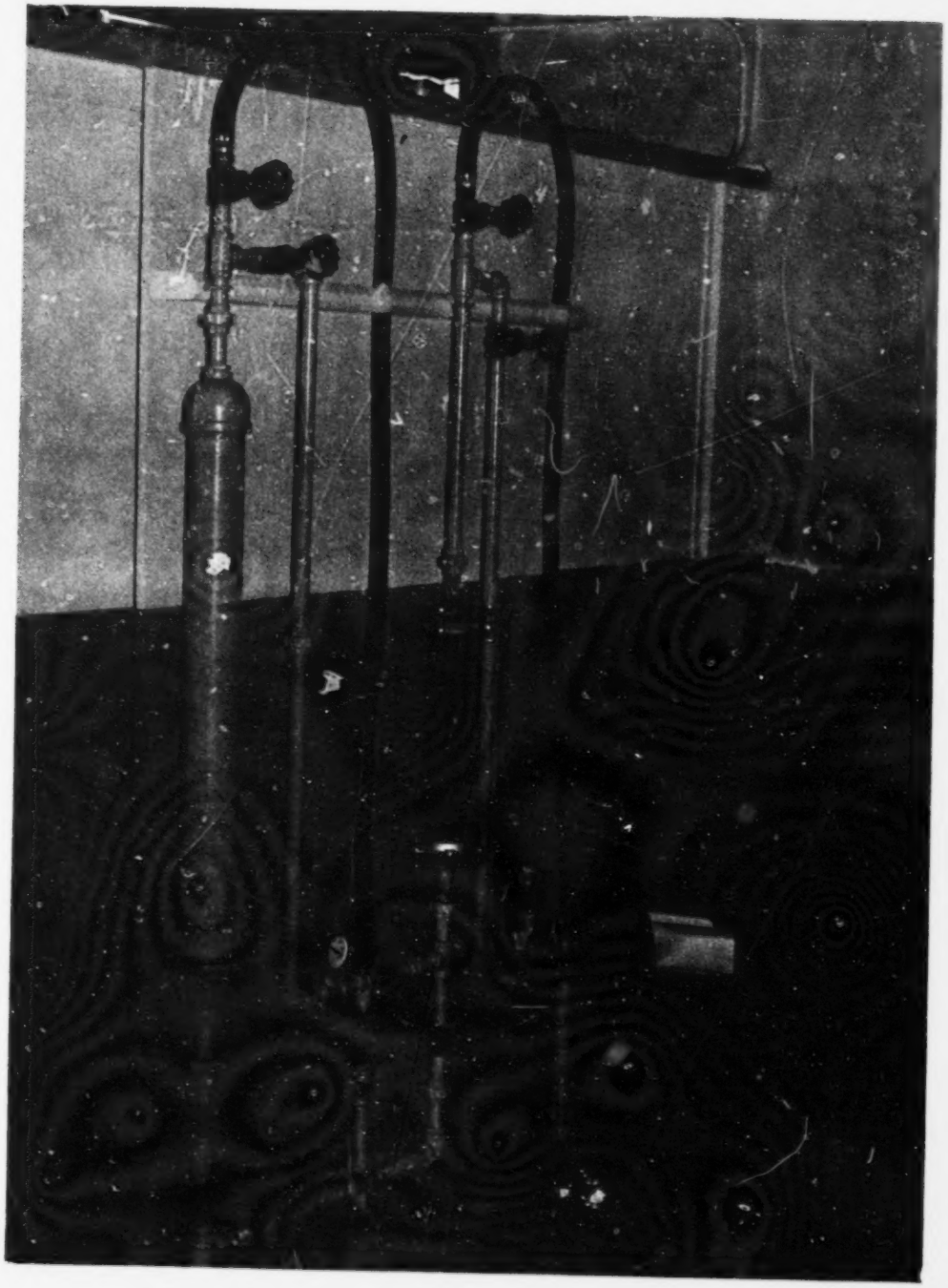

Figure 4 - Typical Carbon Filter Installation.

Reproduced from best available copy.

A water meter, provided by the Public Health Service upon request, located at the end of the system, is used to measure the volume of water sampled. A 5/8 inch by 3/4 inch disc-type meter registering in gallons and capable of measuring flows as low as 1/4 gallon per minute, is best suited for this purpose. A valve following the meter serves to throttle the flow, if necessary. A flow-regulating device may also be used for this purpose.

Fine carbon dust washes out of the filter when it is first started. A few gallons of water should be passed through the top connection and through the carbon filter drain before cutting in the meter, to keep the meter free of the carbon. Do not backwash the carbon. If it becomes clogged, consider the sample run completed and return the filter to the Public Health Service.

The system outlined is not intended to remove all traces of turbidity from the water before passing through the carbon filter. Its purpose is to take out gross materials, most large organisms and permit the required volume of water to pass through the carbon.

Occasionally, some stations may experience clogging of the sand filter due to high algae concentrations. These organisms tend to penetrate into the sand and are difficult to backwash. When this situation is anticipated, an increase in backwashing frequency is indicated.

Backwashing every 2 hours is not uncommon under these circumstances. If, even after this precaution is taken, the sand filter becomes clogged, the only alternative is to replace the sand.

PUMPING

If adequate pressure is not available for sampling raw water, it will be necessary to pump through the filter system. It is important that the pump used does not contaminate the sample through oily packing or other sources. New pumps are sometimes grease-coated and should be thoroughly cleaned before being put into service. If necessary, a pump will be provided upon request. Before a new pump is put into service, it should be thoroughly flushed with hot water containing a little detergent. The pump should be operated against the minimum amount of head required to pass the water through the filter. This may require by-passing of part of the flow.

COLLECTION OF SAMPLE

Water should be passed through the filter at a rate of 1/4 to 1/2 gallon per minute until up to 5000 gallons have been filtered. With highly turbid waters clogging may occur earlier. Although a suitable sample can sometimes be obtained with several hundred gallons, it is desirable to filter a minimum of 2000 gallons, if at all possible.

Since the aim is to get a quantitative measure of organics in water, it is very important to have accurate flow measurements. If difficulties of flow occur this should be noted on the log sheet provided with each filter. Meter readings should be recorded daily on log sheets, and should be designated either in GALLONS or CUBIC FEET. Compute the total volume filtered and enter in the space provided on the record sheet.

PRECAUTIONS

The purpose of the carbon filter is to adsorb small amounts of organic impurities from the water. It is important to avoid contamination of the carbon from other organic sources. Hence the following precautions should be observed:

a. New galvanized fittings are usually coated with oil or grease. The oil should be removed by washing in kerosene followed by a detergent wash before such fittings are used for making connection to the filter.

b. Ordinary organic pipe jointing compounds should not be used. Red lead (lead oxide) mixed to a paste with water can be used for this purpose. (A small supply of red lead is furnished with the filter.)

c. Plastic hose is to be avoided, and if rubber hose is used in any connections it should be flushed thoroughly before being connected to the filter. Copper tubing is ideal for connections.

SHIPPING

When the sampling period has been completed, disconnect the filter and drain off the excess water. Cap both ends and place filter in the shipping box provided. Enclose the record of the filter operation in the box. Ship the filter via Railway Express - Charges Collect - to the address given on page 7.

SECTION IV

COLLECTION OF SAMPLES FOR RADIOACTIVITY MEASUREMENTS

A one-liter grab sample of raw water is collected on a weekly basis for radioactivity measurements.

The sample should be collected at the same time that a portion is taken for the chemical analysis. It should be taken from the stream or from the raw water intake line, prior to any treatment whatever within the plant. This sample must reflect river quality. Therefore, it should be taken before there is any opportunity for sedimentation. Rinse the bottle with raw water before filling it to the bottle neck. Be sure to fill out the enclosed tag and attach it to the bottle. The sample bottle and shipping container are shown in Figure 5.

Figure 5 - Bottle and Shipping Container
For Water Samples For Radioactivity.

SECTION V

COLLECTION OF SAMPLE FOR PLANKTON ANALYSIS

Each shipping container will contain one large polyethylene bottle. Each bottle will contain 100 ml. of preservative - MERTHIOLATE .08% (thimerosal) plus a trace of Lugol's solution.

Figure 6 - Bottle and Shipping Container for Plankton Sample.

The sample for plankton analysis is collected semi-monthly and must be taken on the same day that the raw water is sampled for the chemical analysis. Fill the bottle to the ring marked below the neck on the bottle. Do not over fill. Do Not Rinse, because this will wash out the preservative. The preservative is perishable and becomes unstable at warm temperatures after a month.

When possible the sample should be taken before the water goes through a pump, and always before any treatment whatsoever, including prechlorination, sedimentation or coagulation. The sample should represent stream conditions. If the sample must be taken from a tap, be sure that the running water represents that from continuous flow from the stream, without any settling in the pipes. The best samples are taken directly from the stream just above the water plant intake.

Labels and tags will be provided with each sample bottle. Be sure the bottle is identified with the information requested.

SECTION VI

MEMBRANE FILTER DELAYED INCUBATION PROCEDURE

The sample for the coliform examination is to be collected each week in a separate, sterile bottle, at the time that the collections are made for the chemical analyses. Examinations will be made by the membrane filter delayed incubation technique. Because of the nature of coliform organisms, it is very important that every effort be made to filter the sample, transfer the membranes to the preservative medium and mail to the SEC without undue delay. Samples of water shipped long distances into a central laboratory do not represent a true picture of the coliform populations present at the time of sample collection, if an undue delay has occurred. Do not collect samples on Thursday, as samples remain in shipment too long for satisfactory analysis.

SUPPLIES

The Public Health Service will provide the necessary filtration equipment to those cooperating laboratories which do not already have it on hand. All other necessary supplies will also be provided by the Service. The filtration apparatus and shipping containers for the samples are shown in Figure 7.

The PHS will provide, at intervals, the supplies listed below for the membrane filter coliform test. When the current supply of most or all of the items is exhausted, please return the shipping container with any unused materials to the SEC.

Each shipping container forwarded to the cooperating laboratory will contain:

M-Endo Broth MF (dehydrated)	1/4 pound
Denatured alcohol (25 ml.)	2 tubes
Sodium benzoate solution [12% aq. (25 ml.)]	2 tubes
Sterile ointment tins (3 tins/pack)	26 packs
Sterile MF filters with absorbent pads (10/pack)	8 packs
Phosphate buffer concentrate (20 ml.)	2 tubes
Mailing cartons	26 each
Addressed franked labels for cartons	26 each
Transmittal forms	26 each

SAMPLING

The procedure for preparation of samples is given below.

Equipment and Materials

a. Filter flask of 1000 ml capacity.
b. Vacuum source (pump or aspirator).
c. Sterile graduates or pipettes for measuring sample.
d. Sterile, phosphate buffered distilled water, 99 ml in each. (In an emergency, sterile dechlorinated tap water may be used as a substitute, although this is not a desirable practice.)
e. Three sterile membranes with ointment tins and three absorbent pads for each sample to be examined.
f. Forceps immersed in pure ethyl (or methyl) alcohol.
g. Medium (see below).

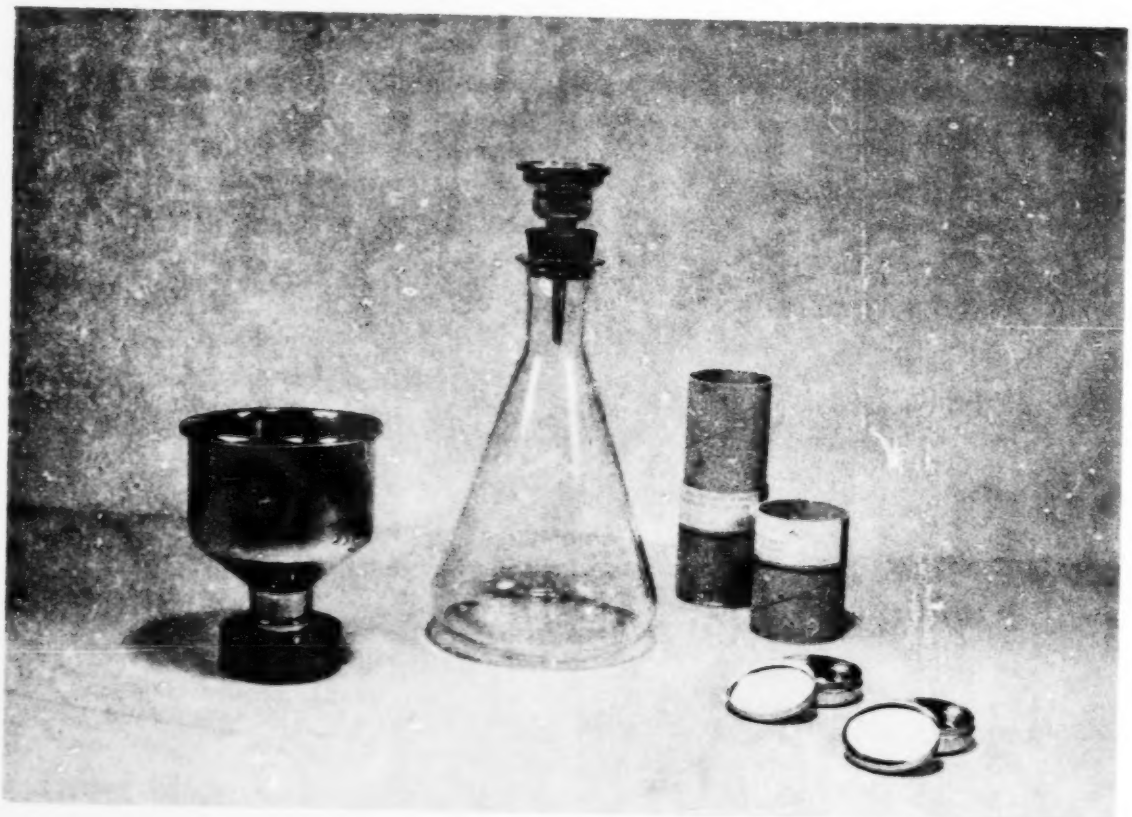

Figure 7 - Filtration Apparatus and Shipping Containers for Coliform Samples.

Preparation of Equipment

a. Wrap funnel and filter base in kraft paper in separate packages and sterilize in autoclave for fifteen minutes at 121°C (15 psi). (For emergency use in absence of sterilization facilities, immerse funnel and filter base in boiling water for five minutes, assemble in the filter flask and draw enough air through porous support disc of filter base to remove excess water. Allow to cool before filtering samples.)

b. Membranes and absorbent pads are supplied by the Public Health Service. Both the membranes and pads are supplied in sterile packets in kraft envelopes or kraft packages and are ready for use in the procedure.

c. Sterile ointment tins in units of three tins are packed in the mailing cartons. The ointment tins are ready for use. Place the absorbent pad in the top of ointment tin - never the bottom.

d. Pipettes, sample bottles, graduates and other equipment should be properly protected by kraft paper and sterilized at 170°C in dry heat for one hour or by steam under pressure (autoclave for 30 minutes at 121°C, 15 psi).

Medium. The medium required for the coliform examination by the delayed incubation membrane filter procedure consists of benzoated M-Endo (preservative) medium.

a. Materials needed for mixing the medium are:

(1) M-Endo Broth - MF - Dehydrated.
(2) Aqueous sodium benzoate solution, 12% (supplied as sterile stock solution in quantity to last six months).
(3) Ethyl alcohol (ethanol or grain alcohol, U.S.P. grade, 95%).
(4) Two sterile 1 ml pipettes.
(5) 100 or 250 ml flask.

b. Directions for preparation of M-Endo Preservative Medium:

(1) Place 50 ml distilled water in a chemically clean 100 to 250 ml Erlenmeyer flask.
(2) Add 1 ml ethyl alcohol (95%).
(3) Add with gentle agitation 2.4 grams dehydrated M-Endo Broth MF.
(4) Heat gently to boiling point to dissolve dehydrated powder (do not boil).
(5) Cool to room temperature.
(6) Add 1.6 ml of sodium benzoate solution and mix gently. This is the finished medium. It will keep 72 hours in a cool, dark place protected from evaporation.

Remarks

(a) The dehydrated medium will absorb moisture rapidly which results in caking and spoiling the powder. The jar containing the dehydrated stock medium must be sealed tightly each time after using to prevent absorption of atmospheric moisture.

(b) The dehydrated medium will be supplied in quarter pound jars to those stations which have available a balance with a sensitivity of ± 0.1 gram.

(c) If no balance is available, the medium can be supplied in small vials containing 2.4 grams each.

(d) For stations which desire to run the immediate MF test, the quantities can be doubled, medium divided in two 50 ml portions in step (5) and 1.6 ml of sodium benzoate solution added to one portion. The portion without added sodium benzoate can be used as a single step procedure for the immediate MF test. (Fifield, C.W. and C.P. Schaufus, JAWWA 1950:50:193-196)

Quantity of Sample for Examination. Successful examination of untreated water for coliform density is dependent upon securing at least one membrane with, preferably, between 20 and 60 coliform colonies and with not more than two or three hundred colonies of all types. Development of colonies from non-coliform flora is generally restricted to a great extent by the indicator in the medium, but there are exceptions. A number of non-coliform colonies will always be present on the membranes except in rare samples where the coliform density numerically exceeds the non-coliform bacteria by many times.

In the delayed incubation membrane filter procedure, the local laboratory does not see the finished membranes to determine conformity with the above requirements. Therefore the central laboratory completing the test will immediately report to the local laboratory when any test is unsatisfactory due to excessive colony density or insufficient number of colonies and, when possible, recommend changes in quantities of water to be filtered for the test.

When there are no existing data on the coliform density of a water sample, the quantities to be used for routine sampling must be determined by a "trial and error" method. In this instance, begin sampling operations by using sample volumes of 0.01, 0.1, and 1.0 ml for the three dilutions. Where reliable coliform data are available from past investigations, the following procedure is recommended for the determination of quantities of water for filtration:

a. Estimate the quantity of water which should contain 20 coliform bacteria (from MPN or other data) and designate this as the "basic test quantity." (BTQ)

b. Filter samples in the order listed below:

(1) One-third the BTQ (one membrane).

(2) The BTQ (one membrane); and

(3) Three times the BTQ (one membrane).

Example: A river on the last examination by a confirmed test had an MPN of 10,000. Coliform density is 10,100/100 or 100 coliforms per ml. The BTQ in ml is obtained by the ratio 20/coliform-density-per ml; giving 20/100 or 0.20 ml. The following quantities would be used as described in Filtration Procedure.

(a) One-third the BTQ or $\frac{0.20}{3}$ or 0.07 ml. (Use 7 ml of a 1-100 dilution for this filtration).

(b) The BTQ of 0.20 ml. (Use 20 ml of a 1-100 dilution or 2 ml of a 1-10 dilution).

(c) Three times the BTQ or 3 X 0.20 is 0.60 ml. (This may be measured directly after additon of about 20 ml of sterile water to the funnel, see f-2 in Filtration Procedure, page 22).

In this example, coliform densities from 3,300 to 85,000 will be determined with one satisfactory membrane containing between 20 and 60 coliform colonies. Although not recommended for routine procedure, under unusual circumstances the coverage could be extended from 1,660 to 142,000 by using counts as low as 10 coliform colonies on the largest quantity or 100 coliform colonies on the least quantity examined.

Filtration Procedure. The membrane filter procedure for coliforms is described on pages 395-404 of Standard Methods for the Examination of Water, Sewage, and Industrial Wastes (10th Edition, 1955) or on pages 508-515 of 11th Edition, 1960. The delayed incubation membrane filter procedure has many procedures in common with the immediate test and special reference is made to the illustration, "Fig. 20, Membrane Filter Assembly" and discussion in paragraph 6, page 397 (10th Ed.) or paragraph 1.6, page 510 (11th Ed.), for a typical assembly of the filtration units. The filtration of the sample after assembly of the equipment is done as follows:

a. Place absorbent pads in sterile ointment tin tops.

b. Saturate each pad with benzoated Endo (preservative) broth.

c. After three or more minutes, remove any excess medium from pads with a sterile pipette.

d. Shake water sample before filtration.

e. Flame forceps and place sterile membrane (grid side up) in filter receptacle and seal funnel on membrane with care in order to avoid injury to the membrane.

f. Measure water sample in funnel with no vacuum on filter flask.

(1) If volume of sample is 10 ml or more, pour measured sample directly on dry membrane.

(2) If between 0.5 ml and 10 ml pour about 20 ml of sterile buffered water in funnel and measure sample into the sterile water in funnel (for good distribution).

(3) If volume of original water sample is less than 0.5 ml, prepare appropriate dilution and proceed as in item (2).

(4) If water sample is:

Over 20 ml,use appropriate sterile graduate.

Between 2-20 ml, use 10 ml or 20 ml sterile pipette.

Between 0.5 - 2 ml, use 1 or 2 ml graduated sterile pipette.

Less than 0.5 ml, make appropriate dilutions in sterile buffered distilled water.

g. Turn on vacuum and filter sample.

h. Rinse sides of funnel down twice with at least 20 ml of sterile buffered distilled water each time, keeping vacuum on.

i. Remove funnel from receptacle and place on sterile paper.

j. Flame the forceps. Using the forceps, remove the membrane from the filter base and lay it on the soaked absorbent pad in the ointment tin cover, grid side up, with a rolling action starting at one edge, as shown in Figure 8. Use care to avoid trapping air bubbles.

k. Place bottom of tin gently over top and proceed with filtration of next quantity of water. All three quantities in ascending order can be filtered in same funnel apparatus with only the rinsing (described in "h") between dilutions.

l. Let membrane stand for about 5 minutes.

m. Inspect each membrane for colorless spots on the MF, which indicate the presence of air bubbles.

n. If the membrane does not have a uniform pink color, remove with forceps and roll on to absorbent pad again.

o. Seal ointment tin by pressing top firmly on.

Figure 8 - Placing Membrane on Pad Soaked with preservative medium.

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SHIPPING

a. Check the identification marking on top of each tin with that recorded on the transmittal sheet. Each ointment tin label should have (1) the date, (2) the station, (3) serial number of filtration, and (4) quantity of water filtered.

b. Place three ointment tins and transmittal sheet in mailing carton for immediate mailing.

c. Samples should be mailed promptly in order to reach Cincinnati before Friday noon. Airmail stickers will be provided to the laboratories where necessary.

SECTION VII

CHEMICAL AND PHYSICAL ANALYSES

Samples taken for chemical and physical analyses should be representative of the raw water in the river. A large enough sample should be collected to satisfy the requirements for the chemical analyses to be performed. A special sampling technique will be required for the dissolved oxygen and B.O.D. determinations and will be described under these tests. The collected sample should be kept in a closed container except when portions are withdrawn for the various analyses. A 2-liter bottle of Pyrex or polyethylene will be satisfactory for a container.

Those analyses affected by time should be performed as soon as possible after the sample is collected. This group includes pH, ammonia nitrogen, alkalinity, turbidity, color and dissolved oxygen.

Detailed procedures are presented below for the chemical and physical analyses of the raw water sample. In most instances the methods described follow closely the procedures in Standard Methods for the Examination of Water, Sewage, and Industrial Wastes (10th Ed.1955, 11th Ed.1960). For the sake of uniformity, it is suggested that these procedures be used by all of the cooperating laboratories in the National Water Quality Network. However, the laboratories have a free choice in using any procedure which has been found to be satisfactory, provided that its precision and accuracy are within normally accepted limits. The Reference Samples, as sent out periodically by the PHS, will be one of the tools by which the precision and accuracy of the various analytical methods in use will be measured.

STANDARD REAGENTS

In general, in the procedures which follow, the standardization of reagents is not described. Many of these reagents are procurable as standard solutions from reliable supply houses. Those which cannot be easily obtained will be provided to participants upon request, by the PHS.

TOTAL ALKALINITY. (Standard Methods 11th Ed. p. 44)

1. Collection of Sample. Use the raw water sample collected for the mineral analyses.

2. Apparatus. Conventional titration equipment.

3. Reagents.

   3.1 Standard Sulfuric Acid 0.0200N

   3.2 Indicator Solution (any of the following can be used):

   a. Methyl orange-dissolve 0.5 g in 1 liter distilled water.

   b. Mixed brom cresol green - methyl red - dissolve 0.02 g. methyl red and 0.10 g brom cresol green in 100 ml ethyl alcohol.

   c. Methyl purple - obtainable from Fleisher Chemical Co., Ben Franklin Station, Washington 4, D. C.

   d. If a pH meter is available with extended leads it can be used as the indicator, with endpoint taken at pH 4.6.

4. Procedure.

   4.1 Place 50 - 100 ml sample into an Erlenmeyer flask or porcelain dish.

   4.2 Add 2-4 drops indicator.

   4.3 Titrate with 0.0200N sulfuric acid to the end point.

5. Calculation: (Total alkalinity as Calcium Carbonate, mg/l)

$$\frac{\text{ml standard acid} \times 1000}{\text{ml sample}} = \text{mg/l alkalinity (as } CaCO_3\text{)}$$

CHLORIDE. (Standard Methods - 11th Edition p. 77)

Three satisfactory methods are described in Standard Methods. The Mohr method, detailed below, is the one currently being used by most laboratories.

1. Collection of Sample. Use raw sample collected for the mineral analyses.

2. Apparatus. Conventionnal titration equipment.

3. Reagents.

3.1 Standard sodium chloride solution 0.0141N:
Dissolve 8.243 g NaCl in 500 ml distilled water.
Dilute 50 ml to 1 liter. Each ml contains 0.500 mg Cl.

3.2 Standard silver nitrate solution, 0.0141N:
Dissolve 2.396 g silver nitrate in 1 liter distilled water.
Standardize by the procedure described below and adjust so that each ml is equivalent to 0.500 mg Cl.

3.3 Potassium chromate indicator:
Dissolve 50 g potassium chromate in about 100 - 200 ml distilled water.
Add silver nitrate until a red precipitate appears.
Let stand overnight and filter.
Dilute the filtrate to 1 liter with distilled water.

3.4 Aluminum hydroxide suspension:
This is used for samples containing appreciable color.
Dissolve 125 g potassium or ammonium alum in 1 liter distilled water.
Add ammonium hydroxide slowly, with stirring to precipitate all the aluminum hydroxide.
Let the precipitate settle.
Decant the supernatant water.
Add distilled water, stir, allow to settle, and decant.
Repeat this operation until the supernatant contains no chloride by test with silver nitrate.

4. Procedure.

4.1 Take 100 ml of sample, or aliquot, containing up to 5.0 mg chloride.
Dilute to 100 ml.

4.2 If sample is colored, add about 3 ml aluminum hydroxide suspension, stir well, filter, wash with 10 - 15 ml distilled water.
Transfer to a white porcelain dish or Erlenmeyer flask.

4.3 Add 1 ml indicator.

4.4 Titrate with standard silver nitrate until color changes from yellow to orange red. Record titration.

4.5 Determine the titration blank, using distilled water in place of sample.

5. Calculation.

$$\frac{[\text{ml of silver nitrate (sample)} - \text{ml silver nitrate (blank)}] \times \text{normality (silver nitrate)} \times 35.46 \times 1000}{\text{ml sample taken}} = \text{mg/l Cl}$$

If normality is 0.0141 the calculation is simplified to:

$$\frac{[\text{ml silver nitrate (sample)} - \text{ml silver nitrate (blank)}] \times 500}{\text{ml sample taken}} = \text{mg/l Cl}$$

CHLORINE DEMAND. (Standard Methods, 10th Ed. p 63, 11th Ed. p 83).

The results of the chlorine demand test are dependent to a considerable degree on the procedures and techniques used. Although there are a number of methods used for the evaluation of chlorine demand of water, a uniform procedure must be used where comparison between several stations is desired. Therefore, to demonstrate changes within a river basin, each laboratory must use the identical test procedure. For this reason, the procedure described below is the only one which should be used in reporting chlorine demand results to the Network.

1. Collection of Sample. Use raw water collected for the mineral analyses. You may need several liters; be sure to collect enough water for all of the tests.

2. Apparatus. About 6-10 clean, quart size bottles or flasks.

3. Reagents.

   3.1 Sulfuric acid (reagent grade) 20 ml/liter.
   Add 20 ml of concentrated sulfuric acid to approximately 750 ml distilled water. Dilute to 1 liter.

   3.2 Potassium iodide, (crystals) reagent grade.

   3.3 Starch solution (See D.O. instructions, page 48.

   3.4 Sodium thiosulfate - 0.025N (See D.O. instructions, Use reagent 4.6(b) page 48.

   3.5 Chlorine solution (500 mg/l)

      a. Stock solution - a bottle of clorox contains approximately 5% chlorine (50,000 mg/l).

      b. Working solution (500 mg/l).
      Take 10 ml of (a) and dilute to 1 liter.

4. Standardizing the Chlorine Solution (Reagent 3.5b)

   4.1 Add about 500 ml of distilled water to a titrating flask or bottle.

   4.2 Add 3-4 crystals of reagent 3.2.

   4.3 Add 10 ml of reagent 3.1.

   4.4 Measure accurately 25.0 ml of working solution 3.5b and add to the flask, swirling rapidly during the addition.

4.5 Titrate with reagent 3.4 to a pale yellow.

4.6 Add 5-10 ml of reagent 3.3 (starch solution).

4.7 Titrate carefully to the disappearance of the blue color, adding the reagent dropwise.

5. Calculating the strength of reagent 3.5b

$$\frac{\text{ml of reagent 3.4} \times 0.025 \times 35.5}{\text{ml of working solution taken}} = \text{mg Cl per ml of working solution, Reagent 3.5b}$$

6. Procedure for Chlorine Demand

6.1 Measure out 500 ml of sample into each of 6-10 flasks or bottles.

6.2 Add 1, 2, 3, 4, etc. ml of reagent 3.5b to the respective samples. These should now contain approximately 1, 2, 3 -- etc. mg/l chlorine.

6.3 Set aside at room temperature in the dark for 1 hour.

6.4 After 1 hour, take the first flask; add 3 or 4 crystals of reagent 3.2.

6.5 Add 10 ml reagent 3.1.

6.6 Add 5-10 ml of reagent 3.3.
If blue color is observed, titrate with reagent 3.4 carefully to the end point. If no blue color is observed, repeat with the second flask, etc.

7. Calculation of Chlorine Residual

7.1 Calculate the chlorine residual in the first flask showing a blue color as follows:

$$\frac{\text{ml of reagent 3.4} \times 0.025 \times 35.5 \times 1000}{\text{ml of sample}} = \text{mg/l Cl residual}$$

When the normality of reagent 3.4 is exactly 0.025 and the sample size is 500 ml, this is simplified to:

ml reagent 3.4 X 1.78 = mg/l Cl residual.

7.2 If chlorine residual is less than 1 mg/l, repeat the titration with the next sample in the series. Continue until you find the first one having a residual greater than 1.0 mg/l.

7.3 Hold the remaining samples 24 hours, then repeat the titration on all of the remaining samples.

8. Calculation of Chlorine Demand.

8.1 One hour demand.

a. Calculate the chlorine added to the first sample which showed a residual greater than 1 mg/l, as follows:

ml reagent 3.5b added X mgCl/ml X 2 = mg/l Cl added.

b. Subtract the chlorine residual which was determined for this sample. The result is the 1 hour demand.

8.2 Twenty-four hour demand

a. Following the titrations in 7.3 calculate the chlorine residual for each of the remaining flasks, as in 7.1.

b. If your water has a breakpoint you will notice that the chlorine residuals will rise, then fall, then rise again as the remaining samples are titrated. For calculating the 24-hour demand, use the first sample having a residual above 1 ppm after the second rise (breakpoint).

c. If your water has no breakpoint, calculate the demand on the first sample having a residual above 1 ppm.

COLOR. (Standard Methods, 11th Ed. p.111.)

For this test to be significant, all turbidity must be removed prior to analysis. The method for removal of turbidity is critical since filtration through filter paper will also remove some of the color.

Turbidity can be removed by. (a) centrifuging and (b) by filtration through a membrane filter.

1. Collection of Sample. Use raw water sample collected for the mineral analyses.

2. Apparatus.

   2.1 A series of Nessler tubes containing platinum color standards or

   2.2 Permanent standards for color as provided with the Hellige aqua tester, the Taylor analyser or similar equipment.

3. Reagents. None

4. Procedure.

   4.1 Take 100 ml of raw sample and remove turbidity by centrifuge or through a membrane filter. If the membrane filter is used, allow turbidity to settle for 1 hour, then gently pour the supernatant through the membrane without disturbing the settled matter. This technique will insure a volume of about 50 ml which is necessary for comparison with standards.

   4.2 Transfer the clear sample to the comparator and match with the appropriate standard.

   4.3 Record to the nearest color unit.

   4.4 If color is too high, prepare a dilution using distilled water. Multiply the color value obtained by the dilution factor.

The PHS will provide color standards for this test, if requested. Either the permanent standards used with comparators or the Nessler tube series can be provided, as desired.

HARDNESS. (Standard Methods 11th Ed. p. 132)

Universal acceptance of the complexometric (EDTA) procedures for hardness has practically eliminated the soap procedure for this test. Standard Methods described several alternative methods. The procedure described below has been found to be the most used, but the others described are equally useful and may be substituted if desired.

1. Collection of Sample. Use the raw water collected for the mineral analyses.

2. Apparatus. Standard titration equipment.

3. Reagents.

3.1 Eriochrome Black T - dry mixture.
Weigh out 1 gram of indicator and thoroughly mix with 250 g of dry sodium chloride crystals.
Keep dry and well stoppered.

3.2 Buffer solution.
Take 570 ml of conc. ammonium hydroxide
Add 67.5 g ammonium chloride
Dilute to 1 liter
Keep well stoppered

3.3 Inhibitor solution.
If an end point is not readily obtainable, it may be due to interference from heavy metals. See p. 115 of Standard Methods 10th Ed. or p. 134, 11th Ed. for the preparation of inhibitor solution to overcome this interference.

3.4 Standard calcium solution.
Dry several grams of pure calcium carbonate at 105°C.
Weigh out 1.00 g into a large Erlenmeyer flask.
Add dilute hydrochloric acid dropwise until the calcium carbonate has dissolved. Add 200 ml H_2O and boil to remove carbon dioxide. Cool. Add methyl red indicator and adjust to orange color with dilute ammonium hydroxide. Transfer completely to a 1-liter volumetric flask and dilute to the mark with distilled water.
1.0 ml of this standard contains 1.00 mg of calcium, as calcium carbonate.

3.5 EDTA titrant.
Dissolve 4.0 g disodium dihydrogen ethylene diamine tetraacetate dihydrate (EDTA) and 0.1 g $MgCl_2 \cdot 6\ H_2O$ in 800 ml water.
Standardize against standard calcium solution as follows:

Put 5.0 ml of solution 3.4 into about 50 ml distilled water. Add 1.0 ml solution 3.2, mix. Add a small scoop of indicator (about .3 - .5 g). Titrate with EDTA to the end point - red to blue. The titration should take between 4.0 and 5.0 ml of titrant. Adjust the EDTA with distilled water so that 1.0 ml equals 1.0 mg hardness as calcium carbonate.
Repeat the titration. If the standard EDTA was correctly adjusted, exactly 5.0 ml of titrant should be used for this titration.
Store in Pyrex or polyethylene only.

4. Procedure.

4.1 Select sample size so that no more than 5.0 ml of titrant is used, and dilute to 50 ml with distilled water.

4.2 Add 1.0 ml buffer solution, reagent 3.2. Mix. Add 1.0 ml inhibitor solution if necessary. Add a scoop of indicator powder, reagent 3.1.

4.3 Titrate with EDTA, reagent 3.5, to blue end point.

5. Calculation.

$$\frac{\text{ml titrant X 1000}}{\text{ml sample}} = \text{mg/l hardness, as calcium carbonate}$$

AMMONIA NITROGEN. (Standard Methods, 11th Ed. p. 167).

The distillation procedure is most accurate where interferences are known to be present and should be used if equipment is available. In the absence of distillation apparatus, the direct procedure can be used and should be satisfactory for most surface waters of the Network. It will be described below.

1. Sample Collection. Use the raw water collected for the mineral analyses.

2. Apparatus.

   2.1 Spectrophotometer or filter photometer, or

   2.2 A Nessler tube series of permanent color standards, as specified on p. 146, Standard Methods, 10th Ed., or p. 172, 11th Ed., or

   2.3 Permanent standards for ammonia nitrogen, as provided with commercial comparators.

3. Reagents.

   3.1 Ammonia free water

      a. Treat distilled water with a slight excess of bromine (about 5-10 mg/l). Let stand overnight. Distill. Discard the first 100 ml. Store distillate in carefully stoppered reservoir to prevent pickup of ammonia from the laboratory air, or

      b. Pass distilled water through a column of mixed resin such as Amberlite Mb-1, or

      c. Add about 10 g of Folin's ammonia permutit to 1 gallon of distilled water and shake.

   3.2 Nessler Reagent

      a. Dissolve 100 g anhydrous mercuric iodide and 70 g anhydrous potassium iodide in about 50-100 ml of ammonia free water.

      b. Prepare a solution of 160 g sodium hydroxide in 500 ml water. Cool.

      Add the first solution (a) to (b) slowly with stirring. Dilute to 1 liter with ammonia free water and store, well stoppered, in pyrex.

3.3 Standard ammonia chloride solution

a. Prepare a stock solution by dissolving 3.819 g anhydrous ammonium chloride in ammonia free water, dilute to 1 liter. 1.0 ml of this solution is equivalent to 1.00 mg of ammonia nitrogen.

b. Dilute 10.0 ml of (a) to 1 liter with ammonia free water. 1.0 ml of this solution is equivalent to 0.01 mg of ammonia nitrogen.

3.4 Rochelle salt solution
Dissolve 500 g of potassium sodium tartrate in 1 liter distilled water. Boil off about 200 ml. Cool. Dilute to 1 liter with ammonia free water.

3.5 Zinc sulfate solution
Dissolve 100 g zinc sulfate in ammonia free water. Dilute to 1 liter.

3.6 Sodium hydroxide
Dissolve about 250 g sodium hydroxide in ammonia free water and dilute to 1 liter.

4. Procedure.

4.1 Take 100 ml of raw water sample. Add 1 ml of zinc sulfate solution, reagent 3.5. Mix.

4.2 Add about 0.5 ml of sodium hydroxide solution, reagent 3.6. pH should be about 10.5 by test.
Mix thoroughly and allow the floc to settle.

4.3 Decant about 25 ml of supernatant through filter paper and discard.
Take the next 50 ml of filtrate (or aliquot diluted with ammonia free water) and add 1-2 drops of Rochelle salt solution, reagent 3.4.

4.4 Add 1.0 ml Nessler reagent, 3.2. Mix. Allow to stand 10 minutes.

4.5 Compare with standards or read in spectrophotometer set at 400-425 wave length.

a. Calibration curve and control test.
Prepare a calibration curve using increasing quantities of standard ammonium chloride solution, reagent 3.3b, into 50 ml of ammonia free water, and carry out the Nesslerization as in steps 4.1 through 4.4. Run a known ammonia sample concurrently with the raw river water whenever the quality of the Nessler reagent is under suspicion.

5. Calculation. Determine the concentration of ammonia nitrogen from the spectrophotometer curve, or comparison with standards. Multiply by the dilution factor, if a dilution was made, and report as mg/l ammonia nitrogen, to the nearest tenth.

HYDROGEN ION CONCENTRATION (pH). (Standard Methods, 11th Ed. p. 193)

pH can be measured either colorimetrically or with a pH meter. The latter measurement is the preferred one although colorimetric measurement is acceptable if a pH meter is not available.

1. Collection of Sample. Use the raw water sample collected for the mineral analyses.

2. Apparatus.

   a. Commercial pH meter, or

   b. Commercial color comparator.

3. Reagents.

   3.1 pH 4 buffer
   Dissolve 10.2 g anhydrous potassium biphthalate in boiled and cooled distilled water. Dilute to 1 liter.

   3.2 pH 7 buffer
   Dissolve 1.361 g anhydrous potassium dihydrogen phosphate and 1.420 g anhydrous disodium hydrogen phosphate in boiled and cooled distilled water. Dilute to 1 liter.

   3.3 pH 9 buffer
   Dissolve 3.81 g sodium tetraborate decahydrate (borax) in boiled and cooled distilled water.

4. Procedure. Follow the recommended procedure of the instrument manufacturer.

5. Results. Report to the nearest 0.1 pH unit.

6. Precautions.

   a. pH meter
   Be sure that electrodes are clean, the glass electrode is not cracked or scratched, the calomel electrode contains saturated potassium chloride solution and its liquid junction (the tip) is not plugged. Remove the rubber stopper on the calomel electrode before use to permit flow of the electrolyte through the tip. When calibrating the instrument use the buffers which will bracket the pH value of the sample.

b. Commercial comparators.
Comparator tubes should be matched, both as to size and color of glass. Be sure that the light passing through the comparator tubes is of equal intensity. If pH reading of sample is at either extreme of the indicator range, check with the next indicator in the series available before reporting pH value. If indicator solution becomes turbid, replace with fresh indicator.

TOTAL DISSOLVED SOLIDS. (Standard Methods, 11th Ed. p. 215)

For this test all turbidity must be removed prior to analysis. Turbidity can be removed as in the Color Test or by filtration. To obtain satisfactory accuracy the sample size selected should be large enough to produce at least 25 mg of residue following the drying operation.

1. Collection of Sample. Use the raw water sample collected for the mineral analyses.

2. Apparatus.

   2.1 Analytical balance, capable of accurate weighing to ± 1 mg.

   2.2 Drying oven controlled at 103 - 105°C.

3. Reagents. None.

4. Procedure.

   4.1 Clean an evaporating dish (about 3"-4" diameter) thoroughly and dry in oven overnight at 103-105°C.

   4.2 Cool and weigh to ± 1.0 mg.

   4.3 Set the evaporating dish in the drying oven or on a steam bath. Measure out a volume of filtered sample which will give at least 25 mg of residue. Add this to the evaporating dish in increments until the total volume selected has been added.

   4.4 Evaporate to dryness and hold for 1 hour at 103°-105°C.

   4.5 Transfer to dessicator and cool.

   4.6 Weigh to ± 1.0 mg.

5. Calculation.

$$\frac{(\text{Wt. of dish + sample - wt. of dish}) \times 1000}{\text{ml of sample taken}} = \text{mg/l total dissolved solids.}$$

SULFATE. (Standard Methods, 11th Ed. p. 237

The gravimetric procedure is the most accurate for samples containing more than 10 mg/l. It is also very time consuming.

The turbidimetric procedure is less accurate than the gravimetric but is much shorter, and is the preferred method for samples up to 60 mg/l. Other methods for sulfate are available and can be used if their accuracy is comparable to the Standard procedures. The turbidimetric method is described in detail below.

1. Collection of Sample. Use the raw water sample collected for the mineral analyses.

2. Apparatus. Commercial photometer or spectrophotometer. Wave length, approximately 420 millimicrons.

3. Reagents.

   3.1 Acid salt solution
   Dissolve 240 g sodium chloride in approximately 900 ml distilled water. Add 20 ml conc. HCl. Dilute to 1 liter.

   3.2 Barium chloride crystals.
   Obtain crystalline $BaCl_2$, 20-30 mesh.

   3.3 Standard sulfuric acid solution 0.02N.
   (Use alkalinity reagent 3.1, p. 26)

4. Procedure.

   4.1 Measure out 50 ml of filtered sample or aliquot diluted to 50 ml if sulfate is above 60 mg/l.

   4.2 Add exactly 10 ml of acid salt solution, reagent 3.1, and mix.

   4.3 Add approximately 0.4-0.5 g barium chloride crystals, reagent 3.2, using a small spoon for uniform measurement, and immediately upon addition of the crystals begin stirring at a constant rate for 1 minute. A magnetic stirrer is very useful for this operation.

   4.4 Let stand 4 minutes.

   4.5 Transfer the suspension to the photometer cell and read immediately. (Prepare a blank by using 50 ml of filtered water to which 10 ml of reagent 3.1 has been added. Use this to zero the photometer.

5. Preparation of Standard Curve.

5.1 Using the sulfuric acid solution, reagent 3.3, add increasing quantities (beginning with zero) up to 60 mg/l, to successive flasks. 1 ml of reagent 3.3 equals 19.2 mg/l when diluted to 50 ml.

5.2 To each flask in the series, carry out procedural steps 4.2 through 4.5 before proceeding with the next flask. Use the zero standard to zero the photometer.

5.3 Prepare a calibration curve, plotting photometer readings vs. concentration of sulfate.

6. Calculation of Results. Read the sulfate concentration of the sample from the calibration curve, multiply by the dilution factor, if any, and report as mg/l sulfate.

TEMPERATURE. (Standard Methods, 11th Ed. p. 270).

Accurate temperature readings are necessary for calculating dissolved oxygen saturation values, for correlation of biological activity and for many other purposes.

The temperature to be reported should be representative of the temperature of the stream at the time the sample is collected. Therefore the temperature must be taken at the sampling point. The thermometer should be immersed in the flowing stream, or in a large sample container filled with the sample and held until the mercury is at rest. The temperature should then be read before removing the thermometer from the sample.

Record the temperature to the nearest fraction of a degree Centigrade which can be estimated from the thermometer being used.

If Fahrenheit thermometer is used, convert readings to Centigrade as follows:

1. Subtract 32 from the Fahrenheit reading.

2. Multiply the result by 10 and divide by 18.

If there is a doubt as to the accuracy of the thermometer being used, it may be sent to the SEC to be checked against a Bureau of Standards thermometer.

TURBIDITY. (Standard Methods, 10th Ed. p. 207, 11th Ed. p. 261).

Turbidity is the expression used to describe the presence of particulate matter such as silt, clay, organic debris, algae or any suspended particle that obstructs the passage of light through water. It is not directly related to the weight of matter in suspension. The Jackson Candle Turbidimeter is the recognized instrument. Other instruments must be calibrated in Jackson units before they can be used in reporting turbidity. Unless turbidity results are reported in Jackson units, they cannot be used by the Network.

1. Collection of the Sample. Use the raw water collected for the mineral analyses.

2. Apparatus.

   2.1 Jackson Candle Turbidimeter, or

   2.2 Commercial turbidimeter, calibrated in Jackson units.

3. Reagents. None.

4. Procedure.

   4.1 For turbidities between 25 and 1000 units

   a. Shake the sample vigorously and add 1-5 ml to the turbidimeter.

   b. Light the candle.

   c. Shake sample again. Add small increments to the turbidimeter until the image of the candle flame is no longer distinguishable. As the sample is added to the tube, the flame outline will gradually fade until it can no longer be distinguished, only diffused light being observed. This is the preliminary reading.

   d. Note the preliminary reading and remove the sample from the tube. Rinse the tube with distilled water.

   e. Shake sample again. Add sufficient sample to the tube to give about 80% of the preliminary reading, then add small increments until the flame disappears again. Record this value.

   f. Repeat this operation twice. Record the average of the three readings.

4.2 For turbidities greater than 1000 units.

a. Make a dilution of the sample such that the turbidity falls near 500 units.

b. Carry out the steps outlined in procedure 4.1.

c. Multiply the average turbidity reading by the dilution factor and record.

4.3 For turbidities below 25 units.

a. Compare the sample with a series of standard turbidities prepared at 5 unit intervals. To prepare the turbidity standards first determine turbidity of a sample of water which falls within the range of the Jackson Candle.

b. Using this water, prepare dilutions with distilled water to give turbidities in the range 5 to 25 units.

c. Transfer these, including the samples, to a series of bottles of identical size, shape and type.

d. Match the sample to the nearest standard.

3.2 Sampling from raw water under pressure. This may be used only if it is known that the raw water is not being aerated during the pumping, and no water treatment has occurred ahead of the point at which the sample is collected.

a. Place a rubber or glass tube from the raw water line into the bottom of a B.O.D. bottle.

b. Run the water into the bottle until it is full and allow it to overflow 2 or 3 times its volume.

c. Slowly withdraw the sampling tube while the water is still running.

d. Carefully stopper the sample bottle, preventing any air from being entrapped.

e. Repeat with the second sample bottle.

4. Reagents.

4.1 Manganous Sulfate. (Use any of the following compounds)

a. $MnSO_4 \cdot 4H_2O$, 480 g

b. $MnSO_4 \cdot 2H_2O$, 400 g

c. $MnSO_4 \cdot H_2O$, 364 g

Dissolve in distilled water and make up to 1 liter.

4.2 Alkaline Iodide - Azide solution.

Dissolve 500 g sodium hydroxide (NaOH)

135 g sodium iodide (NaI)

10 g sodium azide (NaN_3)

all together in distilled water. Make up to 1 liter.

4.3 Conc. sulfuric acid - reagent grade.

4.4 Standard bi-iodate solution. Weigh out 0.8124 g of potassium bi-iodate and dissolve in 1 liter of water. This must be done carefully. The accuracy of the D.O. test depends on the care with which this solution is prepared.

4.5 Starch solution.

a. Make a paste of about 5-6 g potato, arrowroot or soluble starch in a small quantity of water.

b. Add it to about 1 liter of boiling distilled water; boil a few minutes.

c. Cool, allow to stand overnight.

d. Pour off and save the clear solution.

e. Add a few drops of toluene as a preservative.

4.6 Standard thiosulfate solution.

a. Stock solution - 1.0N

a.1 Dissolve 248g sodium thiosulfate (reagent grade) in freshly boiled and cooled distilled water.

a.2 Dilute to 1 liter.

a.3 Add 5 ml chloroform or 1 g sodium hydroxide as a preservative.

b. Titrating solution - 0.025N.
Dilute 25.0 ml of the stock solution with distilled water to 1.0 liter. Add preservative as above.

4.7 Standardization of titrating solution 4.6b.

a. Dissolve about 2g sodium or potassium iodide in 100-150 ml distilled water.

b. Dilute 1 ml of reagent 4.3 to 10 ml with distilled water. Add this to the iodide solution.

c. Add exactly 20.0 ml of reagent 4.4.

d. Dilute to 200 ml and titrate with solution 4.6b until a pale yellow color is reached.

e. Add solution 4.5 and continue the titration carefully to the disappearance of the blue color.

f. If solution 4.6b is of proper strength, it should take exactly 20.0 ml to reach the endpoint.

g. Adjust this reagent, either with 4.6a or distilled water until its strength matches that of reagent 4.4.

h. Recheck this solution once a week and adjust to proper strength.

5. Procedure.

5.1 Add 2 ml of reagent 4.1 followed by 2 ml of reagent 4.2 to the D.O. sample. Take care to add these reagents with the tip of the pipette below the water surface. Carefully stopper and mix vigorously. Do not allow air to be entrapped below the stopper.

5.2 Let stand for 2-3 minutes and repeat the mixing, then let stand until the precipitate in the bottle has settled at least half way.

5.3 Add 2 ml of reagent 4.3 and stopper carefully to prevent air bubbles from entering the bottle. Rinse the outside of the bottle with tap water; then mix thoroughly as before.

5.4 Remove 204 ml of treated sample and transfer to a 500 ml Erlenmeyer flask.

5.5 Titrate with reagent 4.6b until a pale yellow color is reached.

5.6 Add approximately 5 ml of reagent 4.5 and carefully continue the titration to the disappearance of the blue color.

5.7 Record the titration.

5.8 Add 1 drop of reagent 4.4. This should bring back the blue color. If more than one drop is necessary, count the drops and subtract 0.05 ml from the titration for each drop in excess of one.

6. Calculation. If reagent 4.6b is of proper strength, the volume added in the titration above gives the D.O. value directly in mg/l. Record to the first decimal.

BIOCHEMICAL OXYGEN DEMAND (B.O.D.)

1. Collection of Sample. Collect at least 1 liter of water at the same time the D.O. samples are collected. The water in the D.O. sampler may be saved for this purpose.

2. Procedure.

   2.1 Determine the D.O. on one of the samples collected for this purpose.

   a. If the D.O. value is between 9.2 and 6.9 mg/l, place the duplicate D.O. sample in the 20°C incubator and incubate for 5 days. Be careful to maintain the water seal on the sample during the incubation period, then proceed with 2.6 below.

   b. If the D.O. value is above 9.2 or below 6.9, discard the duplicate D.O. sample, and proceed with 2.2.

   2.2 Using the water collected at the time the D.O. sample was taken, let it stand (or warm it) in the laboratory until it reaches room temperature.

   2.3 Shake the sample vigorously to bring it to saturation with atmospheric oxygen. During the shaking the bottle should have air space above the sample to allow the transfer of oxygen. Siphon it carefully into duplicate D.O. bottles.

   2.4 Determine the initial D.O. on one bottle.

   2.5 Incubate the other bottle for 5 days as in 2.1a above.

   2.6 After 5 days of incubation determine the D.O. on the incubated sample.

3. Calculation. Calculate B.O.D. as follows:

   Initial D.O. - Final D.O. = mg/l B.O.D.

In the event that the final D.O. is zero, the B.O.D. reported should be indicated as "greater than". If zero D.O. in the incubated bottle recurs frequently, it will become necessary to set up dilutions of the original sample. Should this be necessary, follow the instructions in "Standard Methods" for the preparation of B.O.D. dilutions.

CHEMICAL OXYGEN DEMAND (C.O.D.) (Reduced Strength Procedure for River Waters)

1. Collection of Sample. Use the raw water collected for the B.O.D. analyses. Be sure to collect enough water for all of the tests.

2. Apparatus. Every precaution should be taken to insure that the sample bottles and other equipment used in this test are clean and free from organic matter.

   2.1 Reflux condenser, Friedrichs (Corning #2600) with ground glass standard taper joint 24/40.

   2.2 300 or 500 ml round bottom flask (Corning #4320) with ground glass neck to fit the condenser; standard taper 24/40.

3. Reagents.

   3.1 Sulfuric acid (reagent grade) concentrated.

   3.2 Ferroin indicator solution.
   Dissolve 1.485 g of 1,10-phenanthroline, with 0.695 g. ferrous sulfate · $7H_2O$ in water and dilute to 100 ml.

   3.3 Standard potassium dichromate.

   a. Dissolve 12.2588 g of potassium dichromate ($K_2Cr_2O_7$), previously dried at 103° C for 2 hours, in distilled water and dilute to 1 liter. This is the stock solution, 0.25N

   b. Carefully measure 100.0 ml of the stock solution into a 1-liter volumetric flask. Dilute to 1.0 liter with distilled water. This is the working solution - 0.025N.

   3.4 Standard ferrous sulfate.

   a. Dissolve 98 g ferrous ammonium sulfate · $6H_2O$ in distilled water. Add 20 ml conc. sulfuric acid, reagent 3.1. Cool. Dilute to 1 liter. This is the stock solution.

   b. Take 100 ml of the stock solution in a 1-liter volumetric flask. Carefully add 20 ml of conc. sulfuric acid, reagent 3.1. Cool. Dilute to 1 liter. This is the working solution. It is approximately 0.025N and must be standardized each time before use by the following procedure:

4. Standardization of Ferrous Ammonium Sulfate. (Reagent 3.4).

4.1 Take 25.0 ml of potassium dichromate (reagent 3.3b) and add to about 300 ml of distilled water in a flask.

4.2 Add 50.0 ml of sulfuric acid (reagent 3.1) carefully. Allow to cool.

4.3 Add 8-10 drops of ferroin indicator (reagent 3.2).

4.4 Titrate with ferrous sulfate (reagent 3.4b) to a deep red color. This is a sharp end point if the sample has been cooled properly before adding the indicator.

5. Calculating the Normality of Ferrous Ammonium Sulfate (Reagent 3.4b).

$$\text{Normality} = \frac{\text{ml reagent 3.3b} \times 0.025}{\text{ml reagent 3.4b}}$$

6. Procedure for C.O.D.

6.1 Take 50.0 ml of the well mixed sample and add it to the round bottom flask.

6.2 Add exactly 25.0 ml of potassium dichromate (reagent 3.3b).

6.3 Carefully and with thorough mixing, add 75 ml of conc. sulfuric acid (reagent 3.1).

6.4 Add 8-10 clean glass beads or porcelain chips. attach the flask to the condenser, place over the burner or hot plate and maintain at gently boiling temperature for 2 hours.

CAUTION: Before applying heat, be sure that the acid has been thoroughly mixed in the round bottom flask, and that the condenser water has been turned on.

6.5 Cool. Wash down the condenser with about 25 ml of distilled water, then transfer, with thorough rinsing, to the titration flask. Dilute to about 350 ml with distilled water.

6.6 Add 8-10 drops of indicator, reagent 3.2, to the cooled sample.

6.7 Titrate with ferrous sulfate, reagent 3.4b, to the red end point.

6.8 If no dichromate remains, as when an immediate red color occurs when reagent 3.4 is added, the test must be re-run using a lesser quantity of sample. Repeat the test using 25.0 ml of sample + 25.0 ml distilled water.

7. Calculation for C.O.D.

7.1 Dichromate consumed by digestion

$$25.0 - \frac{\text{ml Reagent 3.4b X 0.025}}{\text{normality of Reagent 3.4b}} = \text{ml dichromate consumed}$$

7.2 $\frac{\text{ml dichromate consumed}}{\text{ml sample}} \times 200 = \text{Gross Demand}$

7.3 Correction for chloride.

mg/l chloride in sample X 0.23 = chloride correction.

7.4 Subtract result in 7.3 from Gross Demand and report as C.O.D. to the nearest mg/l.

8. C.O.D. Samples for the SEC. Space has been provided in the carbon filter shipping container to simplify forwarding of this sample, as shown in Figure 1, page 8. It will be necessary, therefore, to schedule the collection of this sample for the second week of the month. This is necessary in order to avoid errors due to prolonged storage of C.O.D. samples. Add 50 ml of the sample collected for the weekly mineral analyses to the sample bottle in the carbon filter box. Fill out the attached tag and replace in the carbon filter shipping container. This bottle already contains acid. Do not rinse out or spill any of it. Do not send this sample by mail.

DETERMINATION OF PHOSPHATE (Ortho plus Poly)

Phosphate can be determined as three separate entities in the water environment, as ortho, poly and organic. To do this would require considerable laboratory time, as the organic phosphate procedure is long and tedious, and separate tests are necessary to differentiate ortho from poly. In order to collect the information of greatest value to the Network, and to reduce laboratory time to a minimum, it is felt that a single determination combining orthy plus poly phosphate will fulfill the needs of the Network without imposing an excessive burden on the participating laboratory.

1. Collection of Sample. Use the raw water collected for the mineral analyses.

2. Apparatus.

   2.1 Spectrophotometer or filter photometer, for use at 690 millimicrons or

   2.2 Nessler tubes, 50 ml.

3. Reagents.

   3.1 Phenolphthalein indicator - Dissolve 2.5 gms of phenolphthalein powder in 250 ml of ethyl alcohol, add 250 ml of distilled water, then add 0.020N NaOH dropwise to a faint pink color.

   3.2 Sulfuric acid - Add 310 ml of conc. H_2SO_4 slowly to about 600 ml of distilled water. Cool to room temperature and dilute to 1.0 liter.

   3.3 a. Stock standard phosphate solution (0.50 mg PO_4 per ml). Dry a portion of reagent grade potassium dihydrogen phosphate overnight at 103° before use. Dissolve 0.7164 gms of the KH_2PO_4 in distilled water and make up to 1.0 liter.

   b. Working standard phosphate solution (0.005 mg/PO_4 per ml). Dilute 10.0 ml of the stock standard phosphate to 1.0 liter with distilled water. Protect this solution from the light and make up fresh each month.

   3.4 Ammonium molybdate solution - Dissolve 25.0 gms of $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$ in 175 ml of distilled water.

   Add 155 ml of conc. H_2SO_4 slowly to 400 ml of distilled water and cool.

   Add the molybdate solution to the sulfuric acid solution (never in reverse) and dilute to 1.0 liter.

3.5 Stannous chloride solution - Dissolve 2.5 gms of fresh $SnCl_2 \cdot 2H_2O$ in 10 ml of conc. HCl and dilute to 100 ml with distilled water. Filter, or allow to settle, if turbid. This solution is unstable and should be prepared fresh frequently. It may be stabilized by adding several pieces of mossy tin. Store the solution in a refrigerator and avoid unnecessary contact with air.

4. Procedure.

Preliminary Cleaning of Glassware. Because laboratory detergents contain phosphate which cannot be easily rinsed off, a preliminary treatment with phosphate reagents is necessary to remove that adsorbed on the glass surfaces. After the glassware has been properly cleaned it should be reserved for this test only.

4.1 Clean and thoroughly rinse all glassware in the usual manner.

4.2 Before use, treat each container which will come in contact with the sample as follows:

a. Fill each container with distilled water and add 2.0 ml of ammonium molybdate solution, reagent 3.4, and mix.

b. Add 1 ml of sulfuric acid solution, reagent 3.2, and 1 ml of stannous chloride solution, reagent 3.5. Mix thoroughly and allow to stand for 10-15 minutes. Make certain that all internal surfaces of the containing vessels come in contact with the reaction mixture. Discard the reaction mixture and rinse each container with distilled water.

Once this cleaning procedure has been performed the glassware can be reused without repeating the treatment unless it has been cleaned with detergent.

4.3 Preparation of Standards (Photometric Procedure) Observe room temperature. Standards and samples must be run at same temperature, $\pm$ 2°C, for reproducible results.

a. Add increasing volumes of working standard phosphate reagent 3.3b to several vessels (Previously cleaned according to 4.2). Add 1.0 ml of sulfuric acid, reagent 3.2. Make up to 50.0 ml with distilled water.

The following table is suggested as a convenient series for use with a photometer.

ml of soln 3.3b	mg PO_4/50 ml	PO_4, mg/l
0 (Blank)	0	0 (Blank)
0.5	.0025	.050
1.0	.0050	0.10
2.0	.0100	0.20
5.0	.0250	0.50
10.0	.0500	1.00

b. Add 2.0 ml of ammonium molybdate, reagent 3.4, and mix.

c. Add 5 drops (0.25 ml) of stannous chloride solution, reagent 3.5. Mix and allow to stand for 20 ($\pm$ 5) minutes.

d. Read in spectrophotometer at 690 millimicrons or in a filter photometer at 600-700 millimicrons. Use the Blank to zero the instrument.

e. Prepare calibration curve.

4.4 Raw Water Sample (Photometric Procedure)

a. Filter at least 100 ml of the sample to remove turbidity.

b. Place 100 ml of the filtered sample containing not more than 0.050 mg PO_4 or an aliquot diluted to 100 ml in a beaker or an Erlenmeyer flask. Carry a Blank of 100 ml distilled water along with the sample.

c. Omit this step if the pH is below 8.2. Otherwise

c.1 Add two drops of phenolphthalein, reagent 3.1.

c.2 Add sulfuric acid solution, reagent 3.2, dropwise, to discharge the pink color.

d. Add exactly 2.0 ml of sulfuric acid, reagent 3.2, and boil gently for one-half hour. Add a small amount of distilled water if volume drops below 25 ml during the boiling period.

e. Cool, make up to 100 ml with distilled water and adjust to temperature of standard curve.

f. Take 50 ml of the treated sample and add 2.0 ml of ammonium molybdate solution, reagent 3.4, and mix.

g. Add 5 drops (0.25 ml) of stannous chloride, reagent 3.5, mix, and allow to stand for 20 (± 5) minutes for color development.

h. Read in the spectrophotometer, using the distilled water blank to zero the instrument.

4.5 Calculation

$$\text{mg/l } PO_4 = \text{mg } PO_4 \text{ (from standard curve)} \times \frac{1000}{\text{ml sample}}$$

Report to the nearest 0.1 mg/l.

4.6 Procedure for Nessler Tubes - Follow steps a thru e under photometric procedure 4.4.

f. Cool, transfer 50 ml to a Nessler tube.

g. Add increasing volumes of working standard, reagent 3.3b, to a series of 50 ml Nessler tubes (previously cleaned according to Section 4.2). Add 1.0 ml of sulfuric acid, reagent 3.2. Dilute to 50 ml with distilled water. The following table is suggested as convenient for use in Nessler tubes.

ml of soln 3.3b	mg PO_4/50 ml	PO_4, mg/l
0	0	0
0.5	0.0025	0.05
1.0	0.005	0.10
3.0	0.015	0.30
5.0	0.025	0.50

h. Add 2.0 ml of ammonium molybdate, reagent 3.4, to each standard and sample and mix.

i. Add 5 drops (0.25 ml) of stannous chloride, reagent 3.5. Mix, and let stand for 20 (± 5) minutes for color development.

j. Match the color in the sample with the closest standard.

4.7 Calculation

$$\text{mg/l } PO_4 = \text{mg } PO_4 \text{ (in closest matching standard} \times \frac{1000}{\text{ml sample}}$$

Report to the nearest 0.1 mg/l.

5. Notes.

5.1 The concentration of acid in the sample and standards has a direct effect on the amount of color produced. It is, therefore, important that addition of reagent 3.2 be performed accurately.

5.2 The phosphomolybdate blue produced by the reaction is not stable and tends to fade gradually after about 20 minutes. Most accurate results are obtained when time requirements are observed.

5.3 Temperature is another variable which affects the color formation. Samples and standards may be adjusted to room temperature after boiling by allowing to stand for several hours before addition of reagents 3.4 and 3.5.

5.4 If the phosphate concentration in the sample is completely unknown, several dilutions of the sample may be run simultaneously and that aliquot used for final reading which falls near the middle of the standard series.

5.5 Scrupulous care should be observed in cleaning of the glassware prior to use. If color appears in the Blank, or a particular standard appears out of line, phosphate contamination is probably responsible.

5.6 When using the photometric procedure, only one Blank is necessary if the standards and sample are run at the same time.

5.7 The working standard phosphate solution, reagent 3.3b, should be kept in the dark to prevent reduction of phosphate concentration through algal growth.

5.8 In preparing reagent 3.4, the molybdate must be added to the sulfuric acid, never the reverse. Reversing the addition precipitates some of the molybdate, which is then very difficult to dissolve.

SECTION VIII

HANDLING OF ANALYTICAL DATA

REPORTING OF RESULTS

Each cooperating agency is being furnished a supply of Form PHS-2845-1 (Rev.6-59), NATIONAL WATER QUALITY NETWORK REPORT (FIELD), for reporting the results of the determinations performed by its laboratory. These are provided as multiple copy "snapout" forms, with interleaved carbons to simplify reporting of results. The distribution of the data has been modified since these forms were printed. Both the original or "white" copy and the "blue" are now sent to the Sanitary Engineering Center at Cincinnati; the "salmon" copy goes to the PHS Regional Office serving that area; the "yellow" to the respective State agency; and the "pink" is to be retained by the cooperating laboratory. Where other agencies are to be supplied with the results, the Sanitary Engineering Center will be responsible for preparing and distributing additional copies of the data. Franked envelopes are provided for mailing purposes.

The Sanitary Engineering Center will make a similar distribution of the results of the determinations performed in the Cincinnati laboratory, using Form PHS-2845-2 (Rev.6-59), NATIONAL WATER QUALITY NETWORK REPORT (SEC).

Because the data are to be entered promptly on machine-sorted punch cards, the forms have been designed for easy transcription to the permanent records. A fixed code has been established, which cannot accommodate variations in reporting practice. Therefore, in order to avoid errors in the compilation of the data on a current basis, each reporting laboratory is requested to observe the following procedures in tabulating the analyses on the forms provided:

State: Insert the State in which the sampling point is located.

Station Location: Insert the name of the river being sampled, the city or other geographical "landmark" identifying the location, and the river mileage from the mouth to the station, if available.

Laboratory: Insert the name of the laboratory and the person completing the report.

In reporting data, round out the values to the nearest unit shown on the report form. For example, a chloride value of 20.6 mg/l should be reported as 0021 mg/l. The form may be completed with a hard pencil or ball-point pen, or by typing. Whatever the method used, care should be taken to make each copy legible. A sample completed report form is shown on the following page.

DEPARTMENT OF HEALTH, EDUCATION, AND WELFARE
PUBLIC HEALTH SERVICE
DIVISION OF WATER SUPPLY AND POLLUTION CONTROL

PROVISIONAL DATA—SUBJECT TO REVISION

FOR WASHINGTON USE ONLY						
1-2	3-4	5-6	7-8	9-12		
CHECKED BY:				DATE		

STATE	STATION LOCATION
Minnesota	Mississippi River M796.9 (Cairo Base) Lock & Dam No. 3

LABORATORY	DATE SAMPLE TAKEN
Minneapolis-St. Paul Sanitary District - O. E. Bruss	7-26-60

TEST RESULTS

INSTRUCTIONS: All boxes should contain a figure or preceeding zeros. If test is not made, put "X" in every box
EXAMPLE: If D. O is 10.5 mg/l(ppm) enter as follows [1|0.|5]
If Turbidity is 50 units, enter as follows [0|0|5|0]

1. TEMPERATURE	2|5.4 (13 – 15)	*Degrees C. to tenths*
2. DISSOLVED OXYGEN	0|7.6 (16 – 18)	*mg/l to tenths*
3. pH	0|8.4 (19 – 21)	*To tenths*
4. BIOCHEMICAL OXYGEN DEMAND (*B.O.D.*)	0|4.2 (22 – 24)	*mg/l to tenths*
5. CHEMICAL OXYGEN DEMAND (*C.O.D.*)	0|3|1 (25 – 27)	*mg/l in units*
6. CHLORINE DEMAND - 1 HOUR	0|0.8 (28 – 30)	*mg/l to tenths*
7. CHLORINE DEMAND - 24 HOURS	1|2.1 (31 – 33)	*mg/l to tenths*
8. AMMONIA NITROGEN (NH_3-N)	0|0.0 (34 – 36)	*mg/l to tenths*
9. CHLORIDES	0|0|0|9 (37 – 40)	*mg/l in units*
10. ALKALINITY (*as* $CaCO_3$)	1|5|8 (41 – 43)	*mg/l in units*
11. TOTAL HARDNESS (*as* $CaCO_3$)	0|2|1|6 (44 – 47)	*mg/l in units*
12. COLOR	0|4|0 (48 – 50)	*In scale units*
13. TURBIDITY	0|0|5|0 (51 – 54)	*In scale units*
14. SULFATES	0|0|6|1 (55 – 58)	*mg/l in units*
15. PHOSPHATES	0|0.7 (59 – 61)	*mg/l to tenths*
16. TOTAL DISSOLVED SOLIDS	0|3|0|0 (62 – 65)	*mg/l in units*

DISTRIBUTION: Original (White) and Blue Copy to SEC, Cincinnati, Ohio; Salmon Copy to PHS Regional Office; Yellow Copy to State Agency; and Pink Copy for Retention by Laboratory.

PHS-2845-1 REV. 6-59 NATIONAL WATER QUALITY NETWORK REPORT (FIELD)

FORM APPROVED:
BUDGET BUREAU NO 68-R6 34-I

PRESENTATION OF RESULTS

The Water Supply and Pollution Control Division in Washington will process and print all data. This will be done by transferring data from the original sheets to machine-punched cards for listing in chronological order of testing, in such manner that the determinations performed by the cooperating laboratories and by the Sanitary Engineering Center will be coordinated. A national compilation is printed on an annual basis showing the chronological results by station. Since laboratory reports will be machine-punched on cards as received, the annual summary listing can be made available shortly after the results for the "data year" have been received.

Interim reports can be prepared with data listed by geographical area, such as by State or by major and/or minor river basins. The machine tabulation operation is also capable of carrying out statistical analyses of the data such as the development of means, medians, and frequency distributions. Requests for special analyses of the data, and for tabulations covering particular areas, should be made to the Public Health Service Regional Office concerned.